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Sir:

I, Hiroyuki Nakano, a citizen of Japan, hereby declare and state the following:

1. I graduated from Saitama Institute of Technology of Saitama, Japan in 1988 with a degree of Batchelor of Engineering.

2. Since 1988, I have been employed by Technology Development Center of Nitto Electric Works, Ltd., Japan where my present title is Chief Researcher. During my employment therein, I have conducted research work.

3. I am the author of the following publications:

U.S. Patent Application Serial No. 09/748,012

4. Regarding the reason for the filling amount of glass particles of no more than 3% in the U.S. Patent Application Serial No. 09/748,012 (Our Ref. 142US), I conducted experiments to confirm the releasing ability using the actual equipment, under my supervision and control.

5. Testing Experiments

5-1. Construction of Testing Roller

[0001]

[Detailed Description of the Preferred Embodiments]

Referring to Figs. 1 and 2 in the accompanying drawings, a structure and manufacturing method of one embodiment of a fixing roller according to the present invention will now be described in detail.

[0002]

(Outline of A Fixing Apparatus 10)

With reference to Fig.1, a fixing apparatus 10 having a fixing roller as this one embodiment will first be described. The fixing apparatus 10 includes a fixing housing (not shown) secured to a frame of electronic image forming equipment (not shown), e.g. an electronic printer. In this fixing housing, the fixing apparatus 10 also includes a heating roller 12 as the fixing roller of this embodiment, and a pressing roller 14 pressed to the heating roller 12 with a predetermined pressure.

[0003]

As shown in Fig.2, the heating roller 12 includes an aluminum core 16, a primer layer 18 applied on the periphery of the core 16, and a fluororesin top layer 20 (surface layer) applied on the primer layer 18 so as to bonded on the periphery of the core 16 through the primer layer 18. The heating roller 12 is rotatably driven at a predetermined rotational speed by driving means (not shown) and has a halogen lamp 22 as a heat source built-in. In this embodiment, the heating roller 12 is arranged in 30mm of outside diameter.

[0004]

The top layer is comprised of perfluoroalkoxy resin (tetrafluoroethylene-perfluoroalkylvinylether copolymer resin: hereinafter referred to as PFA) as fluororesin material.

[0005]

On the other hand, while the pressing roller 14 is not shown in detail, it includes an iron core having a nickel-plated surface, a cylindrical elastic layer bonded on the periphery of the core 12 through a primer, and a releasing layer having a predetermined thickness and formed of a fluororesin layer covering the peripheral surface of the elastic layer. In this embodiment, the pressing roller 14 is arranged in 30mm of outside diameter.

[0006]

In the aforementioned coating film on the periphery of the core 16 of the heating roller 12, as a feature of the present invention, a structure having the top layer 20 bonded on the peripheral surface of the core 16 through the primer layer 18, or a

two-layer structure composed of the primer layer 18 and top layer 20, is employed, and glass particles 24 are mixed into at least one of the primer layer 18 and top layer 20.

5-2. Manufacturing Method of Testing Roller

[0007]

A first embodiment in which the glass particles 24 are mixed into only top layer 20, a second embodiment in which the glass particles 24 are mixed into only primer layer 18, and a third embodiment in which the glass particles 24 are mixed into both the primer layer 18 and top layer 20 will be described in turn. Before starting these descriptions, a common manufacturing method of the heating roller 12 in the first to third embodiments will now be described.

[0008]

In the manufacturing method of the heating roller 12, the peripheral surface of the core 16 is subjected to a surface treatment including cleaning, and then applied with a primer. For the second and third embodiments, the glass particles 24 are mixed with a primer solution of raw material at a predetermined weight ratio, and uniformly dispersed over the primer solution. Then, the applied primer is subjected to a forced drying at 80 to 100℃ for about 20 to 30 minutes. Resultingly, the primer layer 18 is formed on the peripheral surface of the core 16.

[0009]

Then, a PFA resin is applied on the periphery of the primer layer 18. For the first and third embodiments, the glass particles 24 are mixed with a PFA resin solution of raw material at a predetermined weight ratio, and uniformly dispersed over the resin solution. Then, the applied PFA is subjected to a forced drying at 80 to 100℃ for about 20 to 30 minutes. Resultingly, the top layer 20 is formed on the peripheral surface of the primer layer 18.

[0010]

Lastly, the entire heating roller 12 is subjected to a firing at 360 to 400℃ for about 30 to 40 minutes. Through this firing process, the heating roller 12 is eventually

completed.

[0011]

On the other hand, the method for forming the PFA top layer 20 is not limited to the application as describe above, and may include a method based on covering with a heat contraction tube made of PFA.

[0012]

Now, the first to third embodiments will be described in turn.

(First Embodiment)

In the first embodiment, the glass particles 24 are mixed into only the top layer 20, but not mixed into the primer layer 18, as shown in Fig.3. In the first embodiment, the Model No.MP-910BK Primer made by DU PONT-MITSUI FLUOROCHEMICALS Co., Ltd. was used as a material of the primer layer 18, and applied to form a layer of 10± 5μm thickness. The Model No.510CL PFA Enamel made by DU PONT-MITSUI FLUOROCHEMICALS Co., Ltd. was used as a material of the top layer 20, and applied to form a layer of 10±5μm thickness. Thus, in the first embodiment, a total thickness of the entire coating film combined the primer layer 18 and top layer 20 is up to 30μm.

[0013]

Further, small grain size of glass particles were used as the glass particles 24 mixed into the top layer 20, and prepared in five kinds mixed in 1%, 2%, 3%, 4%, and 5% by weight, and these embodiments are referred as to first embodiment ①, ②, ③, ④, and ⑤ respectively. The small grain size of glass particles 24 are glass particles having a grain size distribution as shown by a diamond-shaped black dot (◆) in Fig.6 and characteristics shown in the following Table 1.

[0014]

Table 1

small grain size	total moisture (%)	electric conductivity (μS/cm)	electric conductivity blank value (μS/cm)	pH	pH blank value	average grain size (μm)
	0.14	2.0	1.0	5.6	5.8	3.0

[0015]

(Second Embodiment)

In the second embodiment, the glass particles 24 are mixed into only the primer layer 18, but not mixed into the top layer 20, as shown in Fig.4. In the second embodiment, the same material as the above first embodiment was used as a material of the primer layer 18, and applied to form a layer of 10±5μm thickness. The same material as the above first embodiment was used as a material of the top layer 20, and applied to form a layer of 10±5μm thickness. Thus, in the second embodiment, a total thickness of the entire coating film combined the primer layer 18 and top layer 20 is up to 30μm.

[0016]

Regular grain size of glass particles were used as the glass particles 24 mixed into the primer layer 18, and mixed in a weight ratio of 30%. The regular grain size of glass particles 24 are glass particles having a grain size distribution as shown by a diamond-shaped black dot (◆) in Fig.6 and characteristics shown in the following Table 2.

[0017]

Table 2

regular grain size	total moisture (%)	electric conductivity (μS/cm)	electric conductivity blank value (μS/cm)	pH	pH blank value	average grain size (μm)
	0.07	3.4	1.0	5.1	5.8	5.1

[0018]

(Third Embodiment)

In the third embodiment, the glass particles 24 are mixed into the primer layer 18 and top layer 20 respectively, as shown in Fig.5. In the third embodiment, the same material as the above first and second embodiments was used as a material of the primer layer 18, and applied to form a layer of 10±5μm thickness. The same material as the

above first and second embodiments was used as a material of the top layer 20, and applied to form a layer of 10±5μm thickness. Thus, in the third embodiment, a total thickness of the entire coating film combined the primer layer 18 and top layer 20 is up to 30μm.

[0019]

Regular grain size of glass particles equal to the second embodiment were used as the glass particles 24 mixed into the primer layer 18, and mixed in a weight ratio of 30%. Further, small grain size of glass particles were also used as the glass particles 24 mixed into the top layer 20 and prepared in five kinds mixed in 1%, 2%, 3%, 4%, and 5% by weight, and these embodiments are referred as to third embodiment ①, ②, ③, ④, and ⑤ respectively.

[0020]

Then, in parallel with fabricating heating rollers of the first to third embodiments, heating rollers of the conventional structure described in the section of Background of the Invention was fabricated, and these were provides as a first to third conventional examples. The heating rollers of the first to third conventional examples were fabricated to have the same structure identical to the above first to third embodiments.

[0021]

(First Conventional Example)

In the heating roller of the first conventional example, the Model No.MP-902BN Primer made by DU PONT-MITSUI FLUOROCHEMICALS Co., Ltd. was used as a material of the primer layer, and applied to form a layer of 10±5μm thickness. The heat contraction PFA tube (Model No: SMT) made by Gunze Ltd. was used as a material of the top layer. The glass particles were mixed into neither the primer layer nor the top layer.

[0022]

(Second Conventional Example)

In the heating roller of the second conventional example, the Model No.EK-1908GY Primer made by Daikin Industries, Ltd. was used as a material of the primer layer, and

applied to form a layer of 10±5μm thickness. The Model No.EK-4800CR PTFE Resin made by Daikin Industries, Ltd. was used as a material of the top layer, and applied to form a layer of 10±5μm thickness. The glass particles were mixed into neither the primer layer nor the top layer.

[0023]

(Third Conventional Example)

In the heating roller of the third conventional example, the Model No.855-300 Primer made by Du Pont de Nemours & Co. was used as a material of the primer layer, and applied to form a layer of 10±5μm thickness. The Model No.855-403 PTFE Resin/PFA mixture made by Du Pont de Nemours & Co. was used as a material of the top layer, and applied to form a layer of 10±5μm thickness. The glass particles were mixed into neither the primer layer nor the top layer.

5-3. Testing Method

[0024]

The first to third embodiments and the first to third conventional examples constructed as describe above were verified in their durability. The verification test for durability will now be described. This durability means both releasing ability and scratch resistance. When both the releasing ability and the scratch resistance were evaluated as more than "Good", the durability was judged as "Good". When either one of or neither of the releasing ability and the scratch resistance was evaluated as "Good", the durability was judged as "Bad".

[0025]

While the releasing ability will be described later in detail, the deterioration of the releasing ability involved in the operation of passing sheets was judged from an offset phenomenon and a contaminated condition on the coating film surface herein. In the evaluation of the releasing ability described below, the mark "○" indicates "Good" condition, the mark "X" indicating "Bad" condition, and the mark "△" indicating better than "X", but "relatively Bad" condition.

[0026]

While the scratch resistance will also be described later in detail, it was judged from a wear level of the coating film caused by contacting object, such as separating claws, involved in the operation of passing sheets. In the evaluation of the scratch resistance described below, the mark "○" indicates "Good" condition, the mark "X" indicating "Bad" condition, and the mark "△" indicating that it was judged as the "Bad" condition after the number of passed sheets exceeded 100k.

[0027]

(Verification Test of Releasing Ability)

In the verification test of the releasing ability, the DiALTA D350 (made by Minolta Co., Ltd., throughput capacity: 36 sheets / minute) was used as a tester.

[0028]

A method for performing the verification test includes the following steps. Using this tester,

(1) First, with passing an A4 size Komine sheet in its lateral position and manually setting in the midpoint of the density control unit, a start sampling is performed to sample each one of black and white solid sheets.

(2) With using a first chart and setting in the auto-control of the density control unit, a first continuous passing of sheets is performed to continuously duplex for 2000 sheets (2k sheets).

(3) With using a second chart and setting in the auto-control of the density control unit, a second continuous passing of sheets is performed to continuously duplex for 500 sheets (0.5k sheets).

(4) After performing the printing operation of the passing of total 2500 sheets, with passing an A4 size Komine sheet in its lateral position and manually setting in the midpoint of the density control unit, each one of black and white solid sheets is sampled, and an accumulated page number and a passing direction of the sheet are written in each backside of these sampled Komine sheets.

(5) With new passing sheet, the above steps (2) to (4) are repeated.

[0029]

In the verification test performed as described above, when an irregularity in gloss, an image defect from dents caused by separating claws, or a sneaking -through phenomenon of toner contacting to separating claws or thermistors was induced in the black solid image obtained at the step (4), it was judged that a defect of black solid image was induced. When an offset phenomenon was induced in the white solid image obtained at the step (4), it was also judged that a defect of white solid image was induced. In the sampling performed for each 5000 sheets (i.e. 2500 sheet of duplex prints = 5k) at the step (4), when it was founded that a defect was induced either in the black solid image or in the white solid image, it was judged that the releasing ability was "Bad" in a check for each 5k.

[0030]

When the defect continuously occurred twice in the check for each 5k, the releasing ability as the durability was conclusively judged as "Bad". Thus, at this point, the verification test was discontinued and the number of passed sheets at this finish point was recorded.

[0031]

When no defect occurred or the defect did not occur continuously in the check for each 5k, the sheet was passed through up to 300 k.

[0032]

(Verification Test of Scratch Resistance)

At the time when the verification test of the releasing ability was completed or the verification test was discontinued due to occurrence of the defect, the verification test of the scratch resistance was carried out. In the verification test of the scratch resistance, the dent amount at the region of the coating film contacted with separating claws was measured. When the dent amount was 7μm or less, it was judged as "Good ○". When the dent amount was deeper than 7μm, it was judged as "Bad X".

[0033]

This dent-amount measuring test was performed using the SURFCOM 575A (a surface geometry measuring device made by TOKYO SEIMITU Co., Ltd.) with 5.0mm of measuring distance, 0.8 mm of cut off, 0.3mm/sec of measuring speed, 2000 times of vertical magnification, and 20 times of horizontal magnification.

5-4. Test Results

[0034]

(Evaluation of Durability)

The evaluation result based on the above measurement result of the releasing ability in conjunction with the evaluation result based on the measurement result of the dent amount is shown in the following Table 3.

[0035]

Table 3

	coating layer construction			durability			
	material of top layer	with or w/o of glass in top layer	with or w/o of glass in primer layer	Releasing ability		Scratch resistance	
1st conventional embodiment	PFA	W/O	W/O	—	100k/OK	X	100k/NG
2nd conventional embodiment	PTFE	W/O	W/O	X	20k/NG	—	20k/OK
3rd conventional embodiment	PFA/ PTFE	W/O	W/O	△	20k/NG	—	20k/OK
1st embodiment ①	PFA	mixed (1%)	W/O	○	300k/OK	○	300k/OK
1st embodiment ②	PFA	mixed (2%)	W/O	○	300k/OK	○	300k/OK
1st embodiment ③	PFA	mixed (3%)	W/O	○	300k/OK	○	300k/OK
1st embodiment ④	PFA	mixed (4%)	W/O	△	100k/NG	—	100k/OK

1st embodiment ⑤	PFA	mixed (5%)	W/O	X	50k/NG	—	50k/OK
2nd embodiment	PFA	W/O	mixed (30%)	—	150k/OK	△	150k/NG
3rd embodiment ①	PFA	mixed (1%)	mixed (30%)	○	300k/OK	○	300k/OK
3rd embodiment ②	PFA	mixed (2%)	mixed (30%)	○	300k/OK	○	300k/OK
3rd embodiment ③	PFA	mixed (3%)	mixed (30%)	○	300k/OK	○	300k/OK
3rd embodiment ④	PFA	mixed (4%)	mixed (30%)	X	100k/NG	—	100k/OK
3rd embodiment ⑤	PFA	mixed (5%)	mixed (30%)	X	50k/NG	—	50k/OK

[0036]

As is apparent from the Table 3, the first conventional example has a good releasing ability due to the PFA top layer but a bad scratch resistance. The second conventional example has a good scratch resistance due to the PTFE top layer but a bad releasing ability. Since the third conventional example has a top layer made of the mixture of PFA and PTFE, it has a good scratch resistance but a relatively bad releasing ability. Overall, its durability could not be judged as "Good".

It is noted that the marks "○", "△", "X", and "—" are used in evaluating the releasing ability and the scratch resistance shown in Table 3 according to the following procedures.

First, for each roller, durability performance (the numbers of durable sheets) is measured using the releasing ability and the scratch resistance as a factor. Then, when either one of the factors is judged as an "NG", the factor is given the evaluation "X" and the measurement operation is discontinued, and the mark "—" is given to the other factor since no evaluation can be given for that factor. Also, if there is no problem, then the measurement operation will be

discontinued with the upper limit of 300k, and the "○" evaluation is given to the both factors.

Particularly, in the first conventional embodiment, the NG occurred in the scratch resistance when 100k sheets were passed. Therefore, the measurement operation was discontinued at that point and the "X" evaluation was given to the scratch resistance. On the other hand, while there was no problem in the releasing ability when the 100k sheets were passed, the mark "—" was given to this factor since the test for passing sheets of 300k was not performed.

Further, in the second conventional embodiment, the NG occurred in the releasing ability when 20k sheets were passed. Therefore, the measurement operation was discontinued at that point and the "X" evaluation was given to the releasing ability. On the other hand, while there was no problem in the scratch resistance when the 20k sheets were passed, the mark "—" was given to this factor since the test for passing sheets of 300k was not performed.

Also, in the first embodiment ①, since there was no problem at all in the releasing ability as well as in the scratch resistance when 300k sheets were passed, the "○" evaluation was given to the both factors.

Further, in the third embodiment ④, the NG occurred in the releasing ability when 100k sheets were passed. Therefore, the measurement operation was discontinued at that point and the "X" evaluation was given to the releasing ability. On the other hand, while there was no problem in the scratch resistance when the 100k sheets were passed, the mark "—" was given to this factor since the test for passing sheets of 300k was not performed.

[0037]

On the other hand, in the case of the first embodiment where the glass particles 24 are mixed only into the top layer 20, all cases from ① to ③ can be judged as extremely good or "○", except for the cases of ⑤ of 5% mixed and ④ of 4% mixed where they are judged as "X" and "△" respectively since the 300k is not reached.

[0038]

Further, in the case of the second embodiment where the glass particles 24 are mixed only into the primer layer 18, it can be judged as good or "○", while in the case of the third embodiment where the glass particles 24 are mixed into both primer layer 18 and top layer 20,

all cases from ① to ③ can be judged as extremely good or "○" except for the cases of ⑤ of 5% mixed and ④ of 4% mixed.

[0039]

(Verification of Suitable Range of Mixing Ratio of Glass Particles determined by the Releasing Ability)

Based on the foregoing, it is determined that the ratio of the glass particles 24 mixed into the top layer 20 forming the outermost layer of the heating roller 12 is most suitable when it is no more than 3% in weight.

This result would have been unexpected based on the disclosure of Matsuyama, which teaches that any glass fiber content of 25% or less is equally effective.

The undersigned declares that all statements made herein of his own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under ' 1001 of Title 18 of the United States Code and that willful false statements may jeopardize the validity of the application or any patent issued thereon.

Hiroyuki Nakano

Hiroyuki Nakano

Signed this 18th day of March, 2004.